Workshop on Scanning Probe Microscopy
November 20, 2000/ 9:00 am to 12:40 pm/ Bldg. 223, Rm B-002

9:00 Opening remarks / Wai Kwok

Chair: Wai Kwok

9:05 Vitalii K. Vlasko-Vlasov (MSD) (20min-talk + 5min-discussion)
Near Field Magneto-Optics for Studies of Nano-scale Magnetic Structures in Superconducting and Ferromagnetic Samples.

9:30 Gary Wiederrecht and **Gregory Wurtz** (CHM) (15min + 5min)
Apertureless Near-Field Scanning Optical Microscopy and Applications to Transient Spectroscopy

9:50 Alex D. Trifunac and V. F. Tarasov (CHM) (12min + 3min)
Time-Resolved Optically Detected Magnetic Resonance

10:05 Nestor Zaluzec (MSD) (20min + 5min)
Scanning Electron Probes in Nanoscience

10:30 Goran Karapetrov (MSD) (12 min + 3min)
Scanning Tunneling Spectroscopy at MSD

10:45 -------------------------------5 minute coffee break-------------------------

Chair: Ken Gray

10:50 Donqi Li (MSD) (12 min + 3min)
UHV SPM for Nanomagnetism

11:05 U. Welp (MSD) (12 min + 3min)
Spin-polarized STM

11:20 Lahsen Assoufid (APS) (12 min + 3 min)
Use of an Atomic Force Microscope as a Metrology Tool for X-ray Optics and Nano Materials.

11:35 Peter Berghuis (MSD) (12 min + 3 min)
A new low temperature SPM for local measurements and lithography

11:50 Hau Wang (MSD) (12 min + 3 min)
Application of SPM in Soft Material Research

12:05 Orlando Auciello (MSD) (12 min + 3 min)
Scanning Probe Microscopy Sdudies of Domain Dynamics in Thin Films of $Pb(Zr_xTi_{1-x})O_3$

12:20 Jan Hessler (CHM) (15 min + 5 min)
New Detectors for Time Resolved SAXS Measurements

12:40 Adjourn

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Vitalii K. Vlasko-Vlasov (MSD)
Near Field Magneto-Optics for Studies of Nano-scale Magnetic Structures in Superconducting and Ferromagnetic Samples.
Principles of the Near Field Scanning Optical Microscopy (NSOM) operation, different designs, and possible applications of the technique will be reviewed. A possibility of using NSOM polarization contrast for imaging magnetic structures will be discussed and a new approach for the local probe magneto-optics will be presented.

Gary Wiederrecht and **Gregory Wurtz** (CHM)
Apertureless Near-Field Scanning Optical Microscopy and Applications to Transient Spectroscopy

Recently, Near-Field Scanning Optical Microscopy (NSOM) has been developed to overcome the spatial resolution limit classically imposed by the diffraction effect in conventional optics. The value of this limit is about $\lambda/2$ where λ is the wavelength of light (Abbe barrier). NSOM operates as a scanning probe microscope like STM and AFM. However, in the case of NSOM, the optical probe-sample interaction is studied in the near-field zone of the sample surface (that is to say at a nanometric distance from the sample surface).

Historically, the first NSOM architecture developed was aperture NSOM. Today, this type of configuration widely uses a metal coated tapered optical fiber as a probe. This fiber defines a sub-wavelength optical aperture at its extremity used to locally illuminate the sample with evanescent waves or to collect the electromagnetic field confined near the surface. The second family, called apertureless NSOM or scattering NSOM, uses a homogeneous sharp tip whose extremity can be viewed as an electromagnetic nanoantenna [1, 2]. This nanoantenna is immersed in the optical near-field zone of the illuminated sample to convert the evanescent field into propagating waves which are far-field detected. In a fundamental point of view the physical processes involved in both configurations are identical and the differences are restricted to technological considerations. Hence, the use of scattering probes should lead to high resolution capability (related to a sharper tip) as well as a large accessible wavelength range [3, 4]. In addition, the use of the already existing probes for AFM or STM is possible. These techniques were successfully used in many applications in the nanometer scale including local spectroscopy of fluorescent samples [5], the detection of confined electromagnetic fields such as localized surface plasmons [6, 7], magneto-optical imaging [8] or the modification of surface properties [9].

We are developing a transient apertureless NSOM from a commercial AFM device (Multimode/Bioscope from Digital Instruments). In this configuration, we use the AFM probe simultaneously as a mechanical (AFM mode) and an optical probe (NSOM mode). Ultimately, short pulse lasers will be coupled to the NSOM apparatus. This microscope will be devoted to the study of localization/delocalization of basic units of charge, energy or spin within organized collections of nanodomains. This is in order to develop and understand new chemical systems and spectroscopies that permit the initiation of localized excitations that can be delocalized through external control and subsequently localized at a specified location.

See for example:

1. Inouye Y. and Kawata S., *Reflection-mode near-field optical microscope with a metallic probe tip for observing fine structures in semiconductor materials.* Optics Communications, 1997. **134**, p. 31-35.
2. Bachelot R., Gleyzes P., and Boccara A.C., *Reflection-mode scanning near-field optical microscopy using an apertureless metallic tip.* Applied Optics, 1997. **36**(10), p. 2160-2170.
3. Knoll B., et al., *Contrast of microwave near-field microscopy.* Applied Physics Letters, 1997. **70**(20), p. 2667-2669.
4. Lahrech A., et al., *Infrared-reflection-mode near-field microscopy using an apertureless probe with a resolution of l/600.* Optics Letters, 1996. **21**(17), p. 1315-1317.
5. Sanchez E.J., Novotny L., and Sunney Xie X., *Near-Field Fluorescence Microscopy Based on Two-Photon Excitation with Metal Tips.* Physical Review Letters, 1999. **82**(20), p. 4014-4017.
6. Specht M., et al., *Scanning plasmon Near-field microscope.* Physical Review Letters, 1992. **68**(4), p. 476-479.
7. Kryukov A. E., Kim Y. -K., and Ketterson J. B., *Surface plasmon scanning near-field optical microscopy.* Journal of Applied Physics, 1997. **82**(11), p. 5411-5415.
8. Grésillon S., et al., *Transmission-mode apertureless near-field microscope: optical and magneto-optical studies.* Journal of Optics A, 1999(1), p. 178-184.
9. Jersch J. and Dickmann K., *Nanostructure fabrication using laser field enhancement in the near-field of a scanning tunneling microscope tip.* Applied Physics Letters, 1996. **68**(6), p. 868-870.

Alex D. Trifunac and V. F. Tarasov (CHM)

Time-Resolved Optically Detected Magnetic Resonance

Ion-recombination and triplet-doublet quenching are magnetic field dependent light-emitting processes. Microwaves can be used to modulate light emission at resonance and obtain optically detected magnetic resonance spectra of short-lived paramagnetic ions. We have carried out such experiments in liquids and in solids (~4 K to RT) using ionizing or photoionizing radiation to initiate the charge separation giving rise to coherent spin pairs. We propose to develop TRODMR to observe few/single molecule processes.

Nestor Zaluzec (MSD)

Scanning Electron Probes in Nanoscience

The high spatial resolution, relatively fast data acquisition, and quantitative nature of scanning electron probe systems make these instruments natural complements to mechanical scanning systems. The capabilities of existing and developing SEP instruments at ANL in the context of nanoscale characterization will be identified.

Goran Karapetrov (MSD)

Scanning Tunneling Spectroscopy at MSD

During last few years scanning tunneling microscopy has been a primary SPM tool for studying superconductivity at ANL. A Low temperature STM (4.2 K) developed at MSD has been used to image physical structure and tunneling spectroscopy in both low-temperature and high-temperature superconductors. Spectroscopic capability offers vortex imaging in high magnetic fields. Some of the results of this earlier research will be highlighted.

Present STM research is being focused on observation of vortices in $NbSe_2$ single crystals with random distribution of topological defects in order to study the effect of pinning by nanosize columnar defects. In the future we plan to upgrade the capabilities of this instrument to study spin-polarized tunneling in magnetic materials as well as tunneling into magnetic/superconducting interfaces. In addition, we plan to incorporate the latest development in MEMS technology and invest into development on novel SPM techniques for atom manipulation.

Donqi Li (MSD)

UHV SPM for Nanomagnetism

The magnetic properties of a nanostructure system may significantly differ from that of the bulk due to altered dimensionality, structure, surface/interface electronic structure, quantum size effects, and domain structures. Recent progress in self-assembled magnetic structures is promising for us to explore the structure of ~20 nm down to atomic lengthscale. For such studies, UHV STM is a crucial tool not only to understand the nanostructure growth and fabrication, but also to image the quantum states responsible for the new magnetic interactions. I will provide a brief overview on our current efforts on self-assembled nanomagnets and the investigations with AFM/MFM in air. Then I will discuss some future research opportunities in nanomagnetism utilizing UHV SPM, including self-assembled growth, imaging quantum states, spin-polarized STM, etc.

U. Welp (MSD)

Spin-polarized STM

The imaging of magnetic structures at the nm-level is a capability that is central to a variety of research programs within the proposed Center for Nanoscale Materials. Owing to the atomic resolution that can be achieved in conventional topographic STM the combination of STM with a spin dependent contrast holds the potential for the ultimate spatial resolution in magnetic imaging on the atomic level. The physical principles underlying spin-polarized STM will be presented. Recently, major advances in implementing these principles into working spin-polarized STMs have been made which will be described. Some applications of the spin-polarized in the study of exchange coupling across interfaces, proximity effects and re-magnetization processes will be discussed.

Lahsen Assoufid (APS)

Use of an Atomic Force Microscope as a Metrology Tool for X-ray Optics and Nano Materials.

Atomic force microscopy (AFM), the most common scanning probe microscopy (SPM) technique, has been used for a wide variety of imaging and characterization since its invention by Bining *et al.* in 1986 [1]. In particular, with a resolution down to subnanometer scale, AFM along with other topographic SPMs, has played an important role as a metrology tool in the semiconductor industry, as well as a characterization instrument in the emerging field of nanoscience and technology. For example, the behavior of micro- and nanodevices can be considerably affected by subnanometer variations in both their topography and dimensions. AFM provides accurate surface topography, roughness, and dimensional measurements with angstrom resolution. Moreover, the microscope can be operated in noncontact mode, which makes it very useful for nondestructive testing and characterization. It is therefore expected to be a valuable tool for the planned Center for Nanoscale Materials (CNM).

Atomic force microscopy can also provide key parameters in the range of spatial periods that are relevant to x-ray optics, such as surface microroughness, peak-to-valley heights, and the power spectral density function. Applications include evaluation of x-ray optical polished and superpolished substrates, characterization of thin film properties, and film thickness. For example, achieving optimal performance of a multilayer requires a deep understanding of the relationship between surface/thin film parameters and x-ray performance. AFM data and correlation with x-ray measurements can help provide an understanding of thin film fabrication.

The metrology laboratory at the Advanced Photon Source houses a ThermoMicroscopes/TopoMetrix Explorer atomic force microscope, which is used primarily as a means of characterizing x-ray optical surfaces. In this presentation, after a brief overview of the Explorer instrument, both current and future applications and activities with AFM at the Advanced Photon Source metrology laboratory will be described.

[1] G. Binnig, C.F. Quate, and Gerber, Phys. Review. Lett. 56, 930, (1986)

Peter Berghuis (MSD)

A New Low Temperature SPM for Local Measurements and Lithography

Many interactions, e.g. magnetic, strain and charge, which determine the properties of complex oxides such as high temperature superconductors and colossal magnetoresistive materials, have an interaction range in the nanometer domain. Therefore, the development of new device architectures with nanoscale spatial resolution for property measurements is highly desirable. Such architectures can be used to study for example the effects of confinement and interactions between closely spaced nano objects. The Scanning Probe Microscope (SPM) offers the ability not only to manipulate materials on very small length scales, but also to observe them, and thus provides a largely untapped potential for nanofabrication. The advantages of SPM techniques over traditional lithography are: (1) the elimination of chemicals associated with resist-based lithography; (2) the potential for atomic resolution; (3) excellent control over the depth and lateral position; and (4) simultaneous observation of the patterned region resulting in excellent alignment of the fabricated structure. These advantages give scanning probe lithography tools a unique potential to fabricate and study the required architectures for property measurements. I will describe a new low temperature SPM and a procedure to fabricate nanowiring for conductivity measurements on nanodots.

Hau Wang (MSD)

Application of SPM in Soft Material Research
Scanning probe microscopy is a relatively new technique yet it has become an essential tool in the research community since its inception in the early eighties. Some of the reasons include its extremely high resolution and sensitivity, ease of operation, moderate cost factor, and the wide variety of measurements that can be done. A brief overview with emphasis on scanning force microscopy will be provided. Literature examples as well as our recent measurements on organic conductors and polymeric materials will be presented.

Orlando Auciello (MSD)
Scanning Probe Microscopy Sdudies of Domain Dynamics in Thin Films of $Pb(Zr_xTi_{1-x})O_3$
The polarization state and polarization reversal in ferroelectric thin films are naturally linked to the arrangement of domains and their transformations. Therefore, direct imaging of domain structures and investigation of their behavior under an applied electric field can provide valuable information for a general understanding of switching phenomena and the role domains play in degradation effects such as polarization fatigue and polarization retention loss in ferroelectric films. We are using scanning force microscopy (SFM) to perform in situ nanoscale imaging of polarization domains in ferroelectric thin films to study polarization reversal and retention loss. We are also investigating domain dynamics and charge distribution using in situ TEM analysis and TEM holography, respectively. Recent results from studies of domain structures at the nanoscale level in ferroelectric $Pb(Zr_xTi_{1-x})O_3$ (PZT) films will be discussed in terms of the basic scientific and technological implications.

Jan Hessler (CHM)
New Detectors for Time Resolved SAXS Measurements
Small-angle X-ray scattering (SAXS) measurements probe the structure of nanoscale materials, length scales below 30 nm. The current detector used for SAXS measurements is a mosaic CCD detector with nine CCDs. Although exposures below one second can be obtained, the time required to transfer information from the detector to a computer is at least 1.2 seconds. We have designed a new detector, which performs the azmuthal averaging normally done with software, that will allow us to perform SAXS measurements with a temporal resolution below 3.6 microseconds. Therefore, *in situ* measurements of fabrication processes can be performed with the temporal resolution needed to identify the fundamental chemistry and physics of the process. In addition, a next generation detector, which is based on a micro-machining process, will be described. Theoretically this detector can have a temporal resolution as low as 10 nanoseconds. Examples of how these detectors can be used to study the fabrication of nanoscale materials will be described.